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research division**

To: Dr. D. Mills
Director of Research

**TRIP REPORT:
XIII ANNUAL MEETING OF THE FEDERATION OF
ANALYTICAL CHEMISTRY AND SPECTROSCOPY SOCIETIES
ST LOUIS, 29 SEPTEMBER - 2 OCTOBER 1986**

Frank E. Bartoszek

This conference dealt with material measurement methods with emphasis on instrumental techniques to perform novel analyses. Various spectroscopic methods such as Nuclear Magnetic Resonance (NMR) and photoacoustic spectroscopy were discussed and applications both to laboratory and industrial measurements were presented.

INTRODUCTION

This conference dealt mainly with new approaches and solutions to various important laboratory and industrial measurement problems. Much of the work discussed was "state of the art" methods for unusual analysis problems and new experimental techniques that either were being developed for specific analysis problems or that had been developed in other fields and were beginning to find application in the field of analytical chemistry. Two of the more interesting topics are described below.

PHOTOACOUSTIC SPECTROSCOPY

The periodic excitation of a sample by absorption of radiation and the subsequent degradation of this energy to translational energy (a sound wave) is the basis of photoacoustic spectroscopy. In its simplest form radiation is incident on a sample (gas, liquid, or solid) and a microphone within the photoacoustic cavity detects the sound wave from the sample. Photoacoustic spectroscopy offers the advantages of high sensitivity--it is a zero measurement technique in that there is no sig-

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nal without a prior absorption. It is a nondestructive technique, that essentially does not require sample preparation, and is mostly without sample contact. It is also useful for indirect absorption measurements of opaque samples and depth profiling of opaque samples. Detection limits, within sample constraints, are typically in the range 0.1 to 0.001 ppb.

Among the newer developments in this area is the mirage effect. In this method, a "probe" laser beam (such as a helium neon laser) is directed parallel to and just above the surface of the sample. The sample is illuminated perpendicularly and the dissipation of the absorbed energy from the sample surface causes a refractive index gradient in the medium above the sample; this in turn causes a deflection of the probe beam and the deflection is measured on a standard detector.

Another refinement is the introduction of Fourier transform photoacoustic spectroscopy, which offers the usual advantages of multiplex signal recording and high throughput. This method has also been used with the mirage effect. It is useful for both remote and *in situ* detection; it has the advantage that it can be used in hostile environments, or for examining massive objects, solid-liquid interfaces, and localized detection. For example, the method can be used for characterizing species adsorbed on surfaces, or detecting moisture uptake by polymer films, or measuring the thickness of subsurface films.

Several methods for signal enhancement were discussed. These include increasing the pump laser efficiency, optimizing the probe beam focus, maximizing the vibration and sound isolation, and using the slowest practical mirror deflection on the Fourier transform instrument. A novel innovation is the use of deflection media other than air. For example, carbon tetrachloride can be used above the sample and this liquid has a larger refractive index change than air. Estimates indicate that the sensitivity can be improved of the order of a thousand-fold. The method itself is useful for spectroelectrochemistry (characterization of reactions on electrode surfaces), studies of surface corrosion, and studies on heterogeneous catalysts. One difficulty with this method is studying samples in the infrared due to the infrared absorptions of most solvents. There is one way around this absorption problem and this is useful for clear solids. Generally the medium is above the solid to be studied and the excitation beam is focused on the front surface. For clear solids, the pump beam is focused on the lower surface and the medium is below the solid. The probe beam is below the solid and is deflected as usual. The method (both front and reverse deflection) is much improved if the probe beam is as close to the surface as possible.

One application of these methods, which was discussed in a subsequent paper, is materials characterization by backscattered pulsed photothermal radiometry. It was found that by analyzing the time variation of the signals that three pieces of information could be obtained--early times in the decay are related to spectroscopic properties of the material, middle times are related to the thickness of the sample, and long time effects are related to the environment and heat sink effects of the substrate. The method has been used for detecting moisture uptake in polymer films.

NUCLEAR MAGNETIC RESONANCE

Several sessions were devoted to the discussion of recent developments in nuclear magnetic spectroscopy. Much work is being done on biologically interesting materials such as lipids and membranes. There was a good deal of emphasis on magic angle spinning, in which the sample is inserted at 54.7° to the magnetic field; the effect of this is to reduce linewidths. Magic angle spinning of solid samples is frequently coupled with cross-polarization techniques, which reduce the coupling between proton and carbon-13 spins.

One newer development was that of flow NMR, in which the solution flows through the NMR cell. Two possible applications have been thought of for this method--one is to use the NMR as a detector for liquid chromatography and the other is as a probe for obtaining fundamental information on flows within tubes (i.e. plug or laminar flow).

OTHER TOPICS

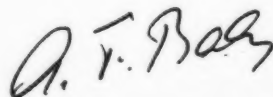
Some sessions were devoted to the use of near infrared reflectance spectroscopy as an industrial tool for on-line quality control measurement. Generally the infrared light from a pulsed laser is sent down a fiber optic to the process stream and the returning signal is measured using time-delay techniques. Several methods to deconvolute more information from these traces were discussed, including derivative modes in which the derivative of the spectrum is examined. By proper choice of wavelengths it is sometimes possible to measure the concentration of one component in the presence of a second, in a wavelength region where the derivative of the second is zero. In addition, this same method can be used to measure small concentrations of a given substance in the presence of a large quantity of interfering matrix. Impurity levels of 0.1% have been successfully measured, even when the spectrum of the matrix is similar to that of the analyte.

In conjunction with other topics, there seems to be considerable interest in the area of computer pattern recognition. Such capability would be useful for computer control of process control and and for the identification of species based on their spectral characteristics.

SUMMARY

The meeting of the Federation of Analytical Chemistry and Spectroscopy Societies presented a broad range of new developments in various areas of laboratory and industrial measurement. Of special interest were new techniques in NMR spectroscopy as well as identifying two possible suppliers of an NMR instrument, which is being purchased by Research Division. In addition, the possibility of using photoacoustic spectroscopy to measure small concentrations of substances is quite exciting.

Approved:



A.F. Baljet
Manager
Electrical Research Department

Submitted:



F.E. Bartoszek
Physical Chemist
Science Section
Electrical Research Department

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